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METHOD FOR ESTIMATION OF RESIDUAL STARCH IN COTTON FABRICS AFTER DESIZING

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Indian Standard

METHOD FOR ESTIMATION OF RESIDUAL STARCH IN COTTON FABRICS AFTER DESIZING

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 20 December 1961, after the draft finalized by the Textile Chemistry Sectional Committee had been approved by the Textile Division Council.

0.2 Starch is applied to warp yarn during the sizing operation, prior to weaving. In order to obtain efficient results in bleaching, dyeing, printing and other finishing treatments, it is essential that starch and other ingredients of size present in grey cloth and also the natural non-cellulosic impurities in cotton are removed completely. Such a removal is accomplished in the processes of desizing and scouring. Desizing is done by steeping the cloth either in water or in dilute mineral acid or more advantageously, by treatment with amylolytic enzymes. Efficient desizing would remove more than 90 percent of the starch present. Starch, if not removed to this extent, will interfere with the efficiency of kiering, yielding degradation products with reducing properties; these products would act detrimentally especially on coloured yarns if they are present in the material under treatment. Hence a routine test to determine the quality of residual starch in the desized cloth is very necessary.

0.3 Wherever a reference to any Indian Standard appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.4 Metric system has been adopted in India and all quantities in this standard have been expressed in this system.

0.5 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960 Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes a method for the estimation of residual starch in cotton fabrics.

2. SAMPLING

2.1 Sample to estimate the amount of residual starch in cotton material in a lot shall be selected so as to be representative of the lot.

2.2 Sample drawn in compliance with an agreement between the buyer and the seller to estimate the starch present in the material in the lot shall be held to be representative of the lot.

3. TEST SPECIMENS

3.1 From the sample under analysis (*see* **2.1** and **2.2**) draw a piece weighing about 10 g. Shred the piece into small bits and mix them thoroughly.

3.2 Draw, from the pieces so shredded, at least five test specimens each weighing about one gram.

4. DETERMINATION OF MOISTURE CONTENT, PERCENT

4.1 Take a test specimen (*see* **3.2**), determine its moisture content by the method prescribed in 5 of *IS : 199-1957 Methods for estimation of moisture, total size or finish, ash and fatty matter in grey and finished cotton textile materials.

4.2 Repeat the procedure on another test specimen (*see* **3.2**).

4.3 Calculate the average of the two values (*see* **4.1** and **4.2**).

5. PREPARATION OF THE EXTRACT

5.1 Take a test specimen (*see* **3.2**). Weigh it accurately. Boil it in about 150 ml of distilled water in a conical flask for about 45 minutes. Cool the contents in the flask and decant the supernatant liquid into a beaker.

NOTE — It would be advantageous to carry out the procedures prescribed in 4 and 5 simultaneously.

5.2 Add about 25 ml of distilled water to the residue in the conical flask and boil it for 30 minutes. Decant the supernatant liquid into the beaker.

5.3 Put a drop of iodine solution on the residue in the conical flask. Observe whether there is any appearance of blue colour.

5.4 Take the residue in the conical flask to be free from all starch if no blue colour is observed (*see* Note).

NOTE — If, however, blue colour is observed, repeat as many times as may be necessary the procedure prescribed in 5.2.

5.5 Filter the decanted liquor twice through a G-3 size sintered glass crucible. Concentrate the filtrate by boiling, cool to room temperature and make up the volume to 100 ml.

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*Since revised.

6. ESTIMATION OF STARCH

6.1 Apparatus

6.1.1 250-*ml Conical Flasks* — two.

6.1.2 *Reflux Condenser.*

6.2 Reagents

6.2.1 *Solution A, Potassium Dichromate Solution* — approximately 0·5 N, but not greater than 0·5 N, containing 24·5 g of potassium dichromate ($K_2Cr_2O_7$) and 300 ml of concentrated sulphuric acid per litre of the solution.

6.2.2 *Solution B, Ferrous Ammonium Sulphate Solution* — slightly greater than 0·1 N, but accurately standardized, containing 39·5 to 40 g of ferrous ammonium sulphate crystals [$FeSO_4 . (NH_4)_2 SO_4 . 6H_2O$] and 20 ml of concentrated sulphuric acid per litre of the solution.

6.2.3 *Phenyl Anthranilic Acid Indicator* — prepared by dissolving 0·13 g of anhydrous sodium carbonate in a small quantity of hot water, and dissolving in this solution 0·26 g of phenyl anthranilic acid and making up the volume to 250 ml.

6.3 Procedure — Transfer 25 ml of the extract (*see* **5.5**) into a 250-ml flask. Add to this, 10 ml of solution *A* (*see* **6.2.1**) and 5 ml of concentrated sulphuric acid. Boil the mixture under reflux for one hour. Cool the contents, make up the volume to 100 ml. Titrate the solution against solution *B* (*see* **6.2.2**) adding 6 to 8 drops of the indicator towards the end of the titration (*see* Note).

NOTE — Appearance of characteristic green colour indicates the completion of the titration.

6.4 Carry out a blank simultaneously following the procedure prescribed in **6.3** but taking 25 ml of distilled water instead of the extract.

6.5 Calculate the oven-dry weight of the specimen by the formula given below:

$$\text{Oven-dry weight, in g,} = w - \frac{w \times m}{100}$$

where

w = weight, in g, of the specimen taken (*see* **5.1**); and

m = moisture content, percent, as calculated (*see* **4.3**).

6.6 Calculate the percentage of starch present in the specimen by the following formula:

$$\text{Percent of starch} = \frac{400 \times (v_1 - v_2) \times N \times 0{\cdot}006\,96}{W}$$

where

v_1 = volume, in ml, of solution *B* required for blank (*see* **6.4**);

v_2 = volume, in ml, of solution *B* required for the extract (*see* **6.3**);

N = normality of solution B; and

W = oven-dry weight, in g, of the specimen as calculated (*see* **6.5**).

NOTE — One millilitre of 1N potassium dichromate corresponds to 0·006 96 g of starch.

6.7 Repeat the procedure prescribed in **5** and **6** with the remaining specimens. Calculate the average of all the values.

7. REPORT

7.1 Report the average as obtained in **6.7** as the percentage of starch present in the sample.

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